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(54) Title: MULTI COMPONENT CONTROLLED RELEASE SYSTEM FOR SANITARY PAPER PRODUCTS

(57) Abstract: The present invention relates to a multi component controlled release system formed of solid hydrophobic nano-particles encapsulated within a moisture-sensitive micro-particle for the controlled release of cosmetic and pharmaceutical active agents in sanitary paper products. This system is useful to control the release of cosmetic and pharmaceutical active agents or countering malodorous compounds upon need and over an extended period of time, in the presence of body fluids. More particularly, the invention pertains to a controlled release system for sanitary paper products that consist of micro particles encapsulating nano particles prepared by a process comprising spray drying a composition of solid nano-particles.

## MULTI COMPONENT CONTROLLED RELEASE SYSTEM FOR SANITARY PAPER PRODUCTS

### Background of the Invention

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#### 1. Field of the Invention

The present invention relates to a multi component controlled release system to be incorporated into sanitary paper products such as; catamenials, diapers sanitary napkins, adult incontinence garments, and other disposable sanitary paper products. More specifically, the invention pertains to cosmetic and pharmaceutical active agents such as fragrances, moisturizing agents, antibacterial compounds, anti-inflammatory agents, and other active agents, encapsulated in a free flowing powder comprising solid hydrophobic nano-particles within a moisture activated micro particle. The free-flowing powder is prepared, preferably, by a process comprising spray drying a composition of solid nano-particles. The controlled release system of the present invention can be useful to control the release of active agents in sanitary paper products, upon need and over an extended period of time, in the presence of body fluids. The system can be also utilized to provide malodor coverage of urine, menses, or body aqueous fluid in general, upon need as well as over an extended period of time. In using malodor counteracting fragrances, the multi component system has the advantage of retaining the volatile constituents of the fragrance until needed, releasing the fragrance upon need (e.g., in the presence of body fluids), and controlling the fragrance release rate to provide malodor coverage over an extended period of time. The controlled release system of the present invention can also be utilized to "signal" the consumer that the product has been soiled and needs to be replaced.

#### 2. Description of the Related Art

Conventional sanitary paper products that are designed to be worn by humans to absorb bodily fluids, such as urine, menstrual fluid, and perspiration are known to acquire a variety of compounds, for example volatile fatty acids (e.g. isovaleric acid), ammonia, amines (e.g. triethylamine), sulphur containing compounds (e.g. mercaptans, sulphides),

alcohols, ketones and aldehydes (e.g. furaldehyde) which release unpleasant odors. These compounds may be present in the bodily fluid or may be produced by fermentation once the bodily fluid is absorbed into the product. In addition, bodily fluids can contain microorganisms that can also generate malodorous by products. Many of these body fluids have an unpleasant odor, or develop such odors when in contact with air and/or bacteria for prolonged periods. Unpleasant odors which emanate from these types of paper products when in use may make the wearer feel uncomfortable and self conscious.

Controlled release systems in sanitary products for odor control have been under investigation for many years. The following types of controlled release systems for sanitary paper products have been disclosed in the literature:

- (i) Porous absorbing materials, such as zeolite, carbon, silica gel and activated alumina;
- (ii) Cyclodextrin inclusion complexes;
- (iii) Porous microcapsules, substantially filled-in structure that are contact-sensitive thereby releasing perfume through the breathable member as the microcapsules receive pressure contact from a user and microcapsules that burst, crush, or rupture to release the malodor counteracting agent; and
- (iv) Microcapsules that comprise of spray dried starch derivatives, natural gums (e.g., gum arabic), and polyhydroxy compounds (mannitol, sorbitol) that quickly release the malodor counteracting agent in the presence of moisture.

Various types of porous systems have been utilized to control malodor in sanitary paper products. US Patent No. 2,690,415 discloses particles of odor-absorbing materials uniformly affixed at the interstices of a permeable web by adhesive to provide an odor absorbent medium, e.g., in catamenials. Particulate carbon, silica gel and activated alumina are used. Shifting and/or displacement of the particulates is avoided and the sheet is flexible.

WO 81/01643 discloses the removal of ammonia (and other toxic or potentially toxic nitrogenous irritants) from diapers by incorporating into the diaper an inorganic aluminosilicate zeolite ammonium ion exchange material. The zeolite is described as synthetic or natural. The zeolite exemplified is naturally occurring clinopholite.

WO 91/11977 discloses a method for decreasing odors associated with bodily fluids comprising contacting the fluids with an odour controlling amount of an intermediate framework  $\text{SiO}_2/\text{AlO}_2$  zeolite.

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US Patent No. 4,525,410 discloses sanitary paper products having antibacterial properties comprising zeolitic particles retaining therein at least one metal ion having bactericidal property and a mixed fibre assembly. The zeolite has a low framework ratio of  $\text{SiO}_2/\text{Al}_2\text{O}_3$ . The zeolite has the ions  $\text{Ag}^+$ ,  $\text{Cu}^{2+}$  or  $\text{Zn}^{+2}$  associated therewith.

10

US Patent No. 6,096,299, discloses an odor control material for decreasing bodily odor comprising of a zeolite having an average particle size (distribution by weight in sieve analysis) of at least 200  $\mu\text{m}$ . The zeolite may optionally be mixed with an absorbent gelling material and/or activated carbon.

15

ABSCENTS (odor-control molecular sieve from Union Carbide) for use in diapers and catamenials are specifically noted in Union Carbide brochure (A. J. Gioffre 1988). The brochure indicates that UC's market research shows potential benefits in such products. US Patent Nos. 4,795,482 and 4,826,497, relate to ABSCENTS used as an odor-controlling agent, generally, and in sanitary products, in particular, and optionally with carbon.

20

Numerous patent literature discusses the application of cyclodextrin complexes for controlling malodor in sanitary paper products. US Patent Nos. 5,429,628, 5,714,445, and 5,660,845 disclose sanitary paper products containing small particle size cyclodextrin for odor control. The invention relates to compositions and articles such as catamenials, diapers, pantliners, paper towels, tissues, underarm shields, etc., which minimize odor caused from body fluids through the incorporation of an effective amount of cyclodextrin, having a particle size of less than 12 microns. Combinations of small particle size cyclodextrins with other odor-controlling materials are also disclosed. The particles provide fast release of the active when the particles are wetted.

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US Patent No. 3,971,852 discloses the use of spray dried particles of starch derivatives, natural gums (e.g., gum arabic), and polyhydroxy compounds (i.e., mannitol, sorbitol) in sanitary paper products. The drawback of these types of materials is the relatively large amount of surface oil, sometimes up to 12%. As a result, the retention of volatile fragrance ingredient may be poor leading to premature leaking of the fragrance. These types of materials, also quickly release the fragrance, or other active ingredients that are encapsulated within their structure upon exposure to water, and would not have the ability to provide malodor coverage, in the presence of body fluids, over an extended period of time.

US Patent No. 5,733,272 discloses a microcapsule for odor control for application in paper product having a positive scent signal which minimizes odor caused by body fluids and which provides a pleasant scent signal to indicate that the odor is being removed. This scent signal is provided by cyclodextrin/perfume inclusion complexes and/or the spray dried microcapsules disclosed in US Patent No. 3,971,852.

US Patents No. 5,591,146, 5,769,832, and 5,769,833 disclose a sanitary napkin having frangible microcapsules located within an adhesive binder that also secures the napkin to a wearer's undergarment. When the release paper that covers the adhesive binder is removed, the microcapsules are crushed or burst and fragrance and/or odor absorbers are released. A capsule wall can be formed of polyvinylalcohol.

US patent No. 5,951,534 also describes a sanitary paper product having one or more touch-sensitive fragrance controlled release systems positioned thereon. Generally, the touch-sensitive fragrance member includes a breathable member fixedly attached to a backing member, such as the outer surface or the backsheet. Release agents are positioned between the breathable member and backing member such that when the breathable member is touched, fragrance is released from the release agents through the breathable member.

A controlled release system for sanitary paper product, such as diapers, based on spray-dried multilamellar phospholipid liposomes is disclosed in US Patent 5,783,211. The liposomes encapsulate a biologically active agent is selected from the group consisting of

anti-inflammatory, antiphlogistic, antibacterial, anti-perspirant, astringent, and anti-fungal agents. The problems with using liposomes and structured vesicles as delivery devices are manifold. These types of systems are unstable, and can only be used for encapsulation of certain types of materials. The liposomes disclosed in US Patent 5,783,211, are released in response to moisture but are not able to sustain the release of the active agents over an extended period of time because liposomes are is not stable in water.

A major challenge in sanitary disposable paper products is to extend the release of active agents and the counteraction of malodor, in the presence of body fluids, over an extended period of time. This problem is acute in overnight products, when a clean sanitary paper product is not readily available, or in nursing homes, where it may take a while until the care provider is available to change the product. Another challenge in sanitary paper products, such as diapers, is the prevention of diaper dermatitis, i.e., diaper rash. To achieve that it is essential to keep the baby's delicate skin dry and free of irritants. Bisabolol is a biologically active agent known to have antiphlogistics and antibacterial properties. This pharmaceutical active agent must be delivered via a specially formulated topical composition. When used in a product in a standard formulation, the whole amount of bisabolol is released at the time of application; so there is an initial burst of the active which quickly decreases below a level that is effective. In order to delay and prolong the release of such fast acting pharmaceutical agents, there is a need for a controlled release system that can provide extended release of the active agent in the presence of body fluids.

Disposable sanitary paper products, such as diapers, adult incontinence briefs, sanitary napkins, and pantliners, because of their convenience and reliability are widely used. While much advancement has been made in the field of disposable products for both infants and adults, a number of problems still exist. Consumers are becoming increasingly educated and expect a high level of sophistication in their sanitary paper products. There is thus a need for an intelligent sanitary paper products which can provide not only physical absorbency of moisture, but also provide prolonged release of active agents, coordinate peak delivery of active agents to a particular biological demand, such as skin wetness, and



counteraction of malodor after the product has been soiled, over an extended period of time, until a new, clean, product is available.

5 A major effort in the art of perfumery has been directed to providing means of treating odors that are offensive to the human sense of smell. In general these products have provided a masking effect by one of two mechanisms. The masking fragrance either suppresses the offensive odor, by providing a more pleasing aroma in large quantities, or blends with the offensive odor to provide a different and more desirable aroma. Unfortunately, in both instances a large amount of fragrance often must be utilized which in  
10 itself often proves to be offensive. Furthermore, the offensive odor is usually still detectable at the levels of masking fragrances that are reasonably tolerable.

#### Summary of the Invention

15 The present invention relates to a multi component controlled release system to deliver cosmetic and pharmaceutical active agents in sanitary paper products upon need, such as in the presence of body fluids, and over an extended period of time. The invention also features a moisture activated controlled release system for sanitary paper products that provides malodor counteracting agents upon need, in the presence of body fluids, and over an  
20 extended period of time.

The invention also provides a free-flowing, powder formed of solid hydrophobic nano-particles of encapsulated cosmetic and pharmaceutical active agents that can be encapsulated in a moisture sensitive micro-particles, characterized by:

- 25 (i) protection of active agents during storage, until needed;  
(ii) moisture triggered release of the active agents upon need in response to body fluids;  
and  
(iii) controlled, continuous release, of effective levels of the active agents in the presence of body fluids over an extended period of time. Pharmaceutical and cosmetic active agents  
30 can be incorporated into hydrophobic solid nano particles. A plurality of the nano particles can be encapsulated in a moisture sensitive matrix to form the micro particles.

Pharmaceutical, cosmetic or malodor counteracting active agents can be incorporated into the nano particle structure, or in both the nano particle structure and the micro particle structure depending on the mechanism and rate desirable for release of the active agents. It has now unexpectedly been found that cosmetic and pharmaceutical active agents, such as fragrances, moisturizing agents, antibacterial compounds, anti-inflammatory active agents, and other active ingredients, encapsulated in nano particles, or dispersed in a nano particle solid matrix, by a process of spray drying, have the ability to sustain the release of these active agents and release them at a controlled rate in the presence of body fluids over an extended period of time.

The invention also provides a free-flowing powder formed of solid hydrophobic nano-particles of encapsulated malodor counteracting fragrance that can be encapsulated in a moisture activated micro-particle characterized by:

- (i) protection of the volatile constituents of the malodor counteracting fragrance during storage, until needed;
- (ii) moisture triggered release of the malodor counteracting fragrance upon need in response to body fluids;
- (iii) controlled, continuous release, of effective levels of the malodor counteracting fragrance in the presence of body fluids over an extended period of time; and
- (iv) high impact fragrance "burst" in response to body fluids that "signal" the consumer that the product has been soiled and needs to be replaced. The present invention has the advantage that the counteraction of malodor over an extended period of time provides the consumer greater confidence in the product performance and a sense of protection, especially in overnight products, when a prompt change of the soiled product is impossible, i.e., when a clean product is not readily available, or in nursing homes where it may take a while for the care provider to change the product.

The invention also provides a method for producing the multi component controlled release system including cosmetic and pharmaceutical active agents that comprise the steps of:

- (i) incorporating the active agents into the solid hydrophobic nano-particles; and
- (ii) forming an aqueous mixture comprising of one or more active agents, the nano-particles, and a water sensitive material, such as, starch derivatives, natural gums, polyvinyl alcohol, proteins, hydrocolloids, or mixture of thereof; and
- 5 (iii) spray drying the mixture to form a dry powder composition.

The invention further provides a process for producing the multi component controlled release system including the cosmetic and pharmaceutical active agents that comprise the steps of:

- 10 (i) heating hydrophobic materials to a temperature above the melting point of the materials to form a melt;
- (ii) dissolving or dispersing a first active agent into the melt;
- (iii) dissolving or dispersing a second active agent and moisture sensitive materials, such as, starch derivatives, natural gums, polyvinyl alcohol, proteins, hydrocolloids, or mixture of
- 15 thereof, in the aqueous phase;
- (iv) heating the composition to above the melting temperature of the hydrophobic material;
- (v) mixing the hot melt with the aqueous phase to form a dispersion;
- (vi) high shear homogenization of the dispersion at a temperature above the melting
- 20 temperature until a homogeneous fine dispersion is obtained having a particle size of from about 1 micron to about 2 microns;
- (vii) cooling the dispersion to ambient temperature; and
- (viii) spray drying the emulsified mixed suspension to form a dry powder composition.

- 25 The incorporation of spray dried nano particles comprising active agents encapsulated within a moisture sensitive matrix in sanitary products was found to extend the release rate of these active and provide malodor coverage after the product has been soiled (in the presence of body fluids), over an extended period of time. In an alternate embodiment, a controlled release composition is formed of hydrophobic nano particles incorporating active agents.
- 30 The invention still further provides a sanitary paper product such as catamenials, diapers, sanitary napkins, pantliners, adult incontinence garments, and other disposable sanitary

paper products comprising the multi component controlled release system or the controlled release system formed of nano particles of the present invention.

#### Detailed Description of the Invention

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The present invention relates to a method of controlling the release of cosmetic and pharmaceutical active agents or counteracting malodorous compounds, over an extended period of time, in the presence of body fluid, using a multi component controlled release system. The invention pertains to cosmetic and pharmaceutical active agents such as

10 fragrances, moisturizing agents, comprising solid hydrophobic nano-particles encapsulated within a moisture sensitive micro particle. The term "particles" is intended to describe solid, substantially spherical particulates. It will be appreciated that other particle shapes can be formed in accordance with the teachings of the present invention. The pharmaceutical and cosmetic active agents can be incorporated into hydrophobic solid nano particles or into a

15 moisture sensitive matrix of the micro particle, or in both the nano and the micro particle structure, depending on the mechanism and rate it needs to be released.

The present invention also relates to a consumer sanitary paper product, such as diapers, catamenials, pantliners, adult incontinence garments, and other disposable sanitary

20 paper products comprising the multi component controlled release system or a controlled release system formed of hydrophobic nano particles of the present invention, which decrease odors associated with bodily fluids such as blood, urine, and the like, and which provides malodor coverage over an extended period of time.

#### 25 I. Cosmetic and Pharmaceutical Active Agents

The controlled release system of the present invention can be useful for the controlled delivery of an extensive array of cosmetic, pharmaceutically or biologically active agents. Suitable active agents include moisturizing agents, fragrances, astringents, anti-

30 inflammatory, anti-microbial, anti-fungal, and the like, which are well know in the art. The

active agents can be encapsulated in the nano particles. The active agents can also be incorporated into the moisture sensitive matrix.

## II. Malodor Counteracting Agents

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Many body fluids have an unpleasant odor, or develop such odors when in contact with air and/or bacteria for prolonged periods. Soiled diapers and other sanitary paper products have a bad, unpleasant malodor. In the context of the present invention the term "malodor" means an unpleasant bad smell caused by urine, feces, blood, sweat, or a combination thereof. The term "counteract" as used herein means the effect on the human sense of smell and/or the malodor resulting in alleviating the offensiveness of the malodor to the human sense of smell. In general, a wide variety of basic approaches for odor control can be used in the present invention. It is not intended that this term be limited to any particular mechanism by which such a result may be obtained.

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A wide variety of basic approaches for odor control can be used in the present invention. Preferably, the approaches for odor control take advantage of knowledge of the compounds responsible for the malodor and the mechanism of odor formation. These include antibacterial to control bacterial growth and fragrances created specifically to blend/mask with the malodors.

20

### II. A. Antibacterial and Bacterial Metabolism

A method for odor control useful in the present invention comprises the use of materials which control bacterial growth and ultimately the bacterial metabolism. Preferred antibacterial compounds of the present invention are bisabolol which has antiphlogistics and antibacterial properties, cetyl pyridinium chloride, zinc chloride, chlorhexidine, quaternary ammonium compounds, parabens, chitin, and pH buffered materials to reduce the pH below the optimum for bacterial metabolism, and the like.

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### II. B. Odor-Neutralizing/Counteracting

The fragrance ingredients and compositions of this invention can be conventional ones known in the art. Selection of any fragrance component, or amount of fragrance, is based on functional and aesthetic considerations. Preferred fragrance components useful in the present invention are fragrance ingredients that are known to function as malodor counteracting. Compositions and methods for counteracting offensive odors which would substantially eliminate such odors without requiring a large amount of fragrance the above-noted disadvantages are particularly desirable. Suitable fragrance ingredients, along with their odor characters, and their physical and chemical properties, are given in "Perfume and Flavor Chemicals (Aroma Chemicals)," Steffen Arctander, published by the author, 1969, incorporated herein by reference. Steffen Arctander, published by the author, 1969, and in "Common Fragrance and flavor Materials – Preparation, Properties and Uses", Kurt Bauer and Dorotea Garbe, published by VCH Verlagsgesellschaft mbH, 1985, incorporated herein by reference. Fragrance components can include highly volatile ingredients having boiling points of about 250°C and moderately volatile ingredients having a boiling point of about 250°C to about 300°C as described in U.S. Patent No. 5,591,146 hereby incorporated by reference into this application. Examples of the highly volatile, low boiling, perfume ingredients are: anethole, benzaldehyde, benzyl acetate, benzyl alcohol, benzyl formate, isobornyl acetate, camphene, cis-citral (neral), citronellal, citronellol, citronellyl acetate, paracymene, decanal, dihydrolinalool, dihydromyrcenol, dimethyl phenyl carbinol, eucalyptol, geranial, geraniol, geranyl acetate, geranyl nitrile, cis-e-hexenyl acetate, hydroxycitronellal, d-limonene, linalool, linalool oxide, linalyl acetate, linalyl propionate, methyl anthranilate, alpha-methyl ionone, methyl nonyl acetaldehyde, methyl phenyl carbinyl acetate, laevomenthyl acetate, menthone, iso-menthone, myrcene, myrcenyl acetate, myrcenol, nerol, neryl acetate, nonyl acetate, phenyl ethyl alcohol, alpha-pinene, beta-pinene, gamma-terpinene, alpha-terpineol, beta-terpineol, terpinyl acetate, and vertenex (para-tertiary-butyl cyclohexyl acetate). Some natural oils also contain large percentages of highly volatile perfume ingredients. For example, lavandin contains as major components: linalool; linalyl acetate; geraniol; and citronellol. Lemon oil and orange terpenes both contain about 95% of d-limonene.

Examples of moderately volatile perfume ingredients suitable for use in the present invention are: amyl cinnamic aldehyde, iso-amyl salicylate, beta-caryophyllene, cedrene, cinnamic alcohol, coumarin, dimethyl benzyl carbinyl acetate, ethyl vanillin, eugenol, iso-eugenol, flor acetate, heliotropine, 3-cis-hexenyl salicylate, hexyl salicylate, lilial (para-  
5 tertiarybutyl-alpha-methyl hydrocinnamic aldehyde), gamma-methyl ionone, nerolidol, patchouli alcohol, phenyl hexanol, beta-selinene, trichloromethyl phenyl carginyl acetate, triethyl citrate, vanillin, and veratraldehyde. Cedarwood terpenes are composed mainly of alpha-cedrene, beta-cedrene, and other  $C_{15}H_{24}$  sesquiterpenes.

10 III. Matrix Materials for Forming the Nano Particles

Suitable solid core materials for forming nano particles of the present invention are inert nontoxic hydrophobic materials with a melting point range between about 45 degrees C and about 120 degrees C. Examples of hydrophobic materials include natural, regenerated,  
15 or synthetic waxes including animal waxes such as beeswax, lanolin and shellac wax, vegetable waxes such as carnauba, candelilla, sugar cane, rice bran, and bayberry wax, mineral waxes such as petroleum waxes including paraffin and microcrystalline wax, and mixtures thereof. Other hydrophobic materials which can be used in the present invention include wax and silicon copolymers, such as candelilla wax and silicone copolymer, ozokrite  
20 wax and silicon copolymers, beeswax and silicon copolymers, and the like. Other hydrophobic compounds which can be used in the present invention include: fatty acid esters such as ethyl stearate, isopropyl myristate, and isopropyl palmitate; high molecular weight fatty alcohols such as cetostearyl alcohol, cetyl alcohol, stearyl alcohol, and oleyl alcohol, solid hydrogenated castor and vegetable oils, hard paraffins, hard fats, and mixtures thereof.  
25 Other hydrophobic compounds which can be used, include triglycerides, preferably of at least food grade purity, which can be produced by synthesis or by isolation from natural sources. Natural sources can include animal fat or vegetable oil, such as soy oil, as a source of long chain triglycerides (LCT). Other triglycerides suitable for use in the present invention are composed of a majority of medium length fatty acids ( $C_{10}$ - $C_{18}$ ), denoted medium chain  
30 triglycerides (MCT). The fatty acid moieties of such triglycerides can be unsaturated or polyunsaturated and mixtures of triglycerides having various fatty acid material. The nano

particle matrix can comprise a single hydrophobic material or a mixture of a plurality of materials. Other hydrophobic materials that are known to those skilled in the art and suitable materials as described in "Industrial Waxes," Vol. I and II, by Bennett F.A.I.C., published by Chemical Publishing Company Inc., 1975 and Martindale, "The Extra Pharmacopoeia", The  
5 Pharmaceutical Press, 28<sup>th</sup>. Edition pp. 1063-1072, 1982 can be used in the present invention.

#### IV. Matrix Materials for Forming a Micro Particle Matrix

Water-sensitive materials for forming the micro particles of the present invention  
10 comprise starch derivatives, polyvinyl alcohol, polysaccharides, hydrocolloids, natural gums, proteins, and mixtures thereof. The polyvinyl alcohol useful in the practice of the invention is partially and fully hydrolyzed polyvinyl acetate, termed "polyvinyl alcohol" with polyvinyl acetate as hydrolyzed to an extent, also termed degree of hydrolysis, of from about 75% up to about 99%. Such materials are prepared by means of any of Examples I-XIV of US Patent  
15 No. 5,051,222 issued on September 24, 1991, the specification for which is incorporated by reference herein.

Polyvinyl alcohol useful for practice of the present invention is Mowiol<sup>®</sup> 3-83, having a molecular weight of about 14,000 Da and degree of hydrolysis of about 83%, Mowiol<sup>®</sup> 3-  
20 98 and a fully hydrolyzed (98%) polyvinyl alcohol having a molecular weight of 16,000 Da commercially available from Gehring-Montgomery, Inc. of Warminster Pennsylvania. Other suitable polyvinyl alcohols are: AIRVOL<sup>®</sup> 205, having a molecular weight of about 15,000-27,000 Da and degree of hydrolysis of about 88%, and VINEX<sup>®</sup> 1025, having molecular weight of 15,000-27,000 Da degree of hydrolysis of about 99% and commercially  
25 available from Air Products & Chemicals, Inc. of Allentown, Pennsylvania; ELVANOL<sup>®</sup> 51-05, having a molecular weight of about 22,000-26,000 Da and degree of hydrolysis of about 89% and commercially available from the Du Pont Company, Polymer Products Department, Wilmington, Delaware; ALCOTEX<sup>®</sup> 78 having a degree of hydrolysis of about 76% to about 79%, ALCOTEX<sup>®</sup> F88/4 having a degree of hydrolysis of about 86% to about 88%  
30 and commercially available from the Harlow Chemical Co. Ltd. Of Templefields, Harlow, Essex, England CM20 2BH; and GOHSENOL<sup>®</sup> GL-03 and GOHSENOL<sup>®</sup> KA-20



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Suitable polysaccharides are polysaccharides of the non-sweet, coloidally-soluble types, such as natural gums, for example, gum arabic, starch derivatives, dextrinized and hydrolyzed starches, and the like. A suitable polysaccharide is a water dispersible, modified starch commercially available as Capule®, N-Lok®, Hi-Cap™ 100 or Hi-Cap™ 200 commercially available from the National Starch and Chemical Company of Bridgewater, New Jersey; Pure-Cote™, commercially available from the Grain Processing Corporation of Muscatine, Iowa. In the preferred embodiment the natural gum is a gum arabic, commercially available from TIC Gums Inc. Belcamp, Midland. Suitable hydrocolloids are xanthan, maltodextrin, galactomanan or tragacanth, preferably maltodextrins such as Maltrin™ M100, and Maltrin™ M150, commercially available from the Grain Processing Corporation of Muscatine, Iowa.

## V. Processing Method

### V.A. Nano Particles

The encapsulated active agent in the nano particles of the present invention can be prepared by the steps of (1) heating hydrophobic materials to a temperature above the melting point to form a melt, (2) dissolving or dispersing the active agent in the melt, (3) emulsifying the melt in the aqueous phase; and (4) cooling the dispersion to ambient temperature to form a fine suspension.

One or more of the pharmaceutical, cosmetic or malodor counteracting agents described above can be incorporated into the hydrophobic solid nano particles. Preferably, about 1% to about 80% of and more preferably about 1% to about 60% by weight of the active agents are used in forming the nano particles.

### V.B. Micro Particles

The controlled release system of the present invention can be prepared by the steps of (a) incorporating selected cosmetic, pharmaceutical, or malodor counteracting active agents into the hydrophobic interior of the nano-particles, (b) forming an aqueous mixture comprising one or more active agents, the nano-particles, and a water sensitive material, and (c) spray drying the mixture of the present invention to form a dry powder composition. Accordingly, the nano particles can be encapsulated into the micro particle structure. One or more of the pharmaceutical, cosmetic or malodor counteracting agents which can be the same or different than the active agents incorporated in the nano particles can be incorporated into the micro particle structure.

A process for producing the multi component controlled release system includes the following stages:

- (i) heating a hydrophobic material to a temperature above the melting point to form a melt;
- (ii) dissolving or dispersing a first active agent into the melt;
- (iii) dissolving or dispersing a second active agent and the water sensitive materials, such as, starch derivative, hydrocolloid, natural gums, polyvinyl alcohol, or mixture of thereof, in the aqueous phase and heating it to above the melting temperature of the hydrophobic material;
- (iv) mixing the hot melt with the aqueous phase to form an dispersion;
- (v) high shear homogenization of the dispersion at a temperature above the melting temperature until a homogeneous fine dispersion is obtained having a particle size of from about 1 microns to about 2 microns;
- (vi) cooling the dispersion to ambient temperature; and
- (vii) spray drying the emulsified mixed suspension to form a dry powder composition.

Homogenization can be accomplished in any suitable fashion with a variety of mixers known in the art such as simple paddle or ribbon mixers although other mixers, such as ribbon or plow blenders, drum agglomerators, and high shear mixers may be used. Suitable equipment for this process include a model Rannie 100 lab homogenizer available from APV Gaulin Inc. Everett, Massachusetts, a rotor stator high shear mixer available from Silverson

Machines, of East Long Meadow, Massachusetts, or Scott Processing Equipment Corp. of Sparta, New Jersey, and other high shear mixers.

The suspension is spray dried to remove the excess water. Spray drying is well known in the art and been used commercially in many applications, including foods where the core material is a flavoring oil and cosmetics where the core material is a fragrance oil. Cf. Balassa, "Microencapsulation in the Food Industry", CRC Critical Review Journal in Food Technology, July 1971, pp 245-265; Barreto, "Spray Dried Perfumes for Specialties, Soap and Chemical Specialties", December 1966; Maleeny, Spray Dried Perfumes, Soap and San Chem, Jan. 1958, pp. 135 et seq.; Flinn and Nack, "Advances in Microencapsulation Techniques", Batelle Technical Review, Vo. 16, No. 2, pp. 2-8 (1967); US patent Nos. 5,525,367; and 5,417,153 which are incorporated herein as references.

In the preferred embodiment, the active agent is present at a level from about 0.01% to about 60%, preferably from about 1% to about 50% by weight of the micro particle. In the preferred embodiment, the nano particles are generally present in the water sensitive matrix at a level from about 1% to about 80%, preferably from about 1% to about 60% by weight of the matrix material with the balance being the active agents and the water sensitive materials. In the preferred embodiment, the moisture sensitive matrix is generally present at a level from about 1% to about 80%, preferably from about 1% to about 60% by weight of the matrix material with the balance being the active agents and the hydrophobic materials.

In one embodiment micro particles are formed by mixing nano particles incorporating a selected active agent with polyvinyl alcohol, or compositions of polyvinyl alcohol and polysaccharides, under conditions sufficient to encapsulate the nano particles. Preferably mixing a selected active agent with the polyvinyl alcohol, or compositions of polyvinyl alcohol and polysaccharides, until the emulsion is formed and then spray drying the emulsion to thereby form an encapsulated nano particle. In the preferred embodiment, the moisture sensitive matrix is formed of a polyvinyl alcohol material at a level from about 1% to about 80%, preferably from about 1% to about 70% by weight of the matrix material with the balance being the amount by weight of active agents and an optimal amount of

polysaccharides. In an alternate embodiment, the polyvinyl alcohol is present in the matrix material in an amount of about 1% to about 80% and the weight of the polysaccharides are present in the amount of about 1% to about 80%. In the preferred embodiment, the active agent composition is generally present at a level from about 0.01% to about 80% preferably from about 1% to about 50% by weight of the encapsulated active agent with the balance being the polyvinyl alcohol or polyvinyl alcohol and polysaccharides. Optionally other conventional ingredients known in the art such as preservatives, surfactants, can be used in accordance with the teachings of the present invention. The multi-component particles of the present invention preferably have size of from about 0.5 micron to about 300 microns, more preferably from about 1 micron to about 200 microns, most preferably from about 2 microns to about 50 microns. The present invention preferably has minimal active agents on the surface of the particles, preferably less than 1%.

Polyvinyl alcohol is an excellent barrier material to the permeation of the volatile fragrance ingredients, and as a result the controlled release systems of the present invention do not provide perceptible odor in the dry state. Upon wetting by a sufficient amount of aqueous fluid such as a body fluid, the matrix can either dissolve to provide a burst of the active ingredients, or swell and soften the matrix to slowly release the encapsulated active agents over an extended period of time, depending on the composition of the matrix, such as the ratio of polyvinyl alcohol to other matrix materials. The use of moisture activated particles which provide varying rates of diffusion are contemplated. For example, the moisture activated particles may diffuse at any of the rates of the following:

- (i) at steady-state or zero-order release rate in which there is a substantially continuous release per unit of time;
- (ii) a first-order release rate in which the rate of release declines towards zero with time; and
- (iii) a delayed release in which the initial rate is slow, but then increases with time.

It has been found that a greater amount of polyvinyl alcohol in the matrix provides slower release rate as compared to a matrix including a lesser amount of polyvinyl alcohol in combination with a polysaccharide. For example, a matrix having about 70% to about 80%

polyvinyl alcohol has a slower release rate than a matrix having about 30% to about 40% polysaccharide and about 40% to about 50% polyvinyl alcohol. For example, if a high amount of polyvinyl alcohol is used in the matrix, such as in the range of about 70% to about 80%, the matrix provides controlled release of the active agent over an extended period of time from the time the matrix contacts moisture up to forty-eight hours. If polyvinyl alcohol is combined with polysaccharide in the matrix, such as in the amount of 30% to about 40% polyvinyl alcohol and 30% to about 40% of polysaccharide, a greater amount of active agent is released upon contact with moisture to provide a "burst" of the active agent and the active agent is released over a shorter period of time for example from the time the matrix contacts the fluid up to the range of about 6 hours to about twenty-four hours. Typically, the active agent at the surface of the particle can be released upon contact with the fluid with the remainder of the active agent being either released in a burst if the matrix dissolves or over an extended period of time upon swelling and softening of the matrix.

Nano particles formed of a hydrophobic material provide a controlled release system in order to release the active agent over an extended period of time by molecular diffusion. Active agents in the hydrophobic matrix of the nano particles can be released by transient diffusion. The theoretical early and late time approximation of the release rate of the active ingredients dissolved in the hydrophobic matrix of the nano particles can be calculated from the following equations:

Early time approximation

$$(m_t/m_{\infty}) < 0.4$$

$$\frac{M_t}{M_{\infty}} = 4 \left( \frac{D_p t}{\pi r^2} \right)^{1/2} - \frac{D_p t}{r^2} \quad (1)$$

$$\frac{dM_t/M_{\infty}}{dt} = 2 \left( \frac{D_p}{\pi r^2 t} \right)^{1/2} - \frac{D_p}{r^2} \quad (2)$$

Late time approximation

$$(m_t/m_{\infty}) > 0.6$$

$$\frac{M_t}{M_\infty} = 1 - \frac{4}{(2.405)^2} \exp\left(\frac{-(2.405)^2 D_p t}{r^2}\right) \quad (3)$$

$$\frac{dM_t/M_\infty}{dt} = 1 - \frac{4D_p}{r^2} \exp\left(\frac{-(2.405)^2 D_p t}{r^2}\right) \quad (4)$$

wherein:

$r$  is the radius of the cylinder,

- 5  $m_\infty$  is the amount fragrance released from the controlled release system after infinite time;  
 $m_t$  is the amount fragrance released from the controlled release system after time  $t$ ; and  
 $D_p$  is the diffusion coefficient of the fragrance or aroma chemical in the matrix

The release rate for releasing the active agents from the hydrophobic nano particles is  
 10 typically slower than the release rate for releasing active agent from the moisture sensitive  
 matrix. The active agents can be selected to be incorporated into either the hydrophobic nano  
 particles or the moisture sensitive matrix depending on the desired time for release of the  
 active agents. For example, a predetermined first active agent can be incorporated in the  
 moisture sensitive matrix to be released upon contact with moisture upon soiling the product  
 15 and a predetermined second active agent can be incorporated in the hydrophobic nano  
 particles for release over an extended period of time during or after the first agent has been  
 released. For example, the moisture sensitive matrix formed in accordance with the present  
 invention can release the first active agent upon contact with moisture to provide a "burst"  
 with continued release of the first active agent up to about twenty-four hours and nano  
 20 particles formed in accordance with the present invention can release the active agent  
 depending on the release rate from an initial time such as within one hour, up to a period of  
 one week.

#### Incorporation of Controlled Release System into Sanitary Paper products

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It is commonly known that when in use, the body fluid is not normally distributed to  
 the whole sanitary paper product, e.g., diaper, but usually localized in a portion of the  
 product. Modern disposable diapers are designed with a concentration of the fluid absorbent  
 material at different locations depending on the sex of the wearers. Similarly, it is not

necessary to apply the particles of the present invention to the entire sanitary paper product. Preferably, the particles are applied to areas most likely to be wetted by body fluids to avoid waste in the areas which normally do not receive the body fluids. Therefore, it is preferred to incorporate the particles of the present invention by uniformly sprinkling, mixing, or  
5 distributing them into the superabsorbent powder that is incorporated onto these preferred locations of sanitary paper products.

The invention can be further illustrated by the following examples preferred  
embodiments thereof, although it will be understood that these examples are included merely  
10 for purposes of illustration and are not intended to limit the scope of the invention unless otherwise specifically indicated. All percentages, ratios, and parts herein, in the Specification, Examples, and Claims, are by weight and are approximations unless otherwise stated.

Preparation of Encapsulated Malodor Counteracting FragranceEXAMPLE 1

A malodor counteracting fragrance composition used in the following examples is as follows:

Perfume Composition	Component ( %Wt.)
Alpha Pinene	5.0
Cedarwood Terpenes	20.0
Dihydro Myrcenol	10.0
Eugenol	5.0
Lavandin	20.0
Lemon Oil CP	10.0
Orange Terpenes	15.0
Phenyl Ethyl Alcohol	15.0

The following procedure is used for the preparation of multi component controlled release system with a malodor counteractive fragrance as the active agent in the hydrophobic phase and cetylpyridinium chloride (CPC), an anti-bacterial active agent in the moisture sensitive phase. The solid hydrophobic nano particles are composed of candelilla wax/silicon copolymer from Strahl & Pitsch Inc. of West Babylon, New-York.

80 grams of candelilla wax/silicon copolymer is placed in an oven at 80 degrees °C and allowed to melt. 600 grams of deionized water are placed into 1 gallon vessel, fitted with a all-purpose silicon rubber heater (Cole-Palmer Instrument Company). 196 grams of polyvinyl alcohol (Mowiol® 3-83, having a molecular weight of about 14,000 Da and degree



of hydrolysis of about 83%, commercially available from Gehring-Montgomery, Inc. of Warminster, Pennsylvania) and 4 grams of CPC are added to the water and the aqueous solution is heated to 90 degrees °C while mixing it with a propeller mixer. The candelilla wax/silicon copolymer melt is removed from the oven and 120 grams of the fragrance are mixed into the wax by hand with a glass rod. The fragrance/wax mixture is poured into the aqueous solution and the dispersion is homogenized at 20,000 psi using a Rannie 100 lab homogenizer available from APV Gaulin Inc. The dispersion is cooled to ambient temperature by passing it through a tube-in-tube heat exchanger (Model 00413, Exergy Inc. Hanson Massachusetts) to form a suspension. The resulting suspension is spray dried with a Bowen Lab Model Drier (at Spray-Tek of Middlesex, New Jersey) utilizing 250 c.f.m of air with an inlet temperature of 380 °F, and outlet temperature of 225 °F and a wheel speed of 45,000 r.p.m to produce a free flowing, dry powder, consisting of 30% malodor counteracting fragrance encapsulated in the solid hydrophobic nano particles and 1% CPC in the water sensitive matrix of the micro particles.

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## EXAMPLE 2

80 grams of cutina wax is placed in an oven at 70 degrees °C and allowed to melt. 600 grams of deionized water are placed into 1gallon vessel, fitted with a all-purpose silicon rubber heater (Cole-Palmer Instrument Company). 100 grams of Hi-Cap™ 100 (commercially available from the National Starch and Chemical Company of Bridgewater, New Jersey) and 100 grams of Maltrin™ M100 (commercially available from the Grain Processing Corporation of Muscatine, Iowa) are added to the water and the aqueous solution is heated to 90 degree C while mixing it with a propeller mixer. Cutina wax is removed from the oven and 120 grams of the fragrance are mixed into the wax by hand with a glass rod. The fragrance/wax mixture is poured into the aqueous solution and the dispersion is homogenized at 20,000 psi using a Rannie 100 lab homogenizer available from APV Gaulin Inc. The dispersion is cooled to ambient temperature by passing it through a tube-in-tube heat exchanger (Model 00413, Exergy Inc. Hanson Massachusetts) to form a suspension. The resulting suspension is spray dried with a Bowen Lab Model Drier (at Spray-Tek of Middlesex, New Jersey) utilizing 250 c.f.m of air with an inlet temperature of 380 °F, and

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outlet temperature of 225 °F and a wheel speed of 45,000 r.p.m to produce a free flowing, dry powder, consisting of 30% malodor counteracting fragrance encapsulated in the solid hydrophobic nano particles and 1% CPC in the water sensitive matrix of the micro particles.

5 INCORPORATION OF THE MULTI COMPONENT CONTROLLED RELEASE  
SYSTEM IN SANITARY PAPER PRODUCTS

### Incorporation of the Spray Dried Powder into a Diapers

10 EXAMPLE 3

The sanitary paper product used in the following examples was a Pamper diaper manufactured by the Procter & Gamble Company, Cincinnati, OH. Each diaper was cut to half and opened.

0.500 grams of the spray dried nano particle powder of examples 1 and 2 was homogeneously dispersed into the absorbent core of the product. The diaper structure was then reconstituted and sealed along the edges by means of adhesive. Three products were prepared for each sample. A commercial, market Pampers diaper and a diaper comprising the neat fragrance (0.15 grams of fragrance oil) were used as a reference.

## TEST METHODS

### Odor Perception Test

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EXAMPLE 4

Odor perception is, by its nature, a very subjective determination. According to the procedure, the samples to be tested are provided to a panel of six odor specialists who independently rank the urine malodor coverage of the samples on a scale of 1 (neutral, the intensity of the malodor is very low, hardly perceived) to 10 (high pleasant odor of the

malodor counteracting fragrance, no malodor is perceived). Samples yielding an odor ranking above about 3.0 possess a malodor, which would hardly be noticed by the general public. Two types of urine malodor were employed in the testing of the systems; reconstituted human urine and synthetic urine solution. The reconstituted urine (reconstituted according to product instructions) is commercially available from Sigma under the trade designation Urinary Metabolite Lyophilizate, from human male urine, Catalog No. U 6378. The synthetic urine is composed of 0.385% by weight urea and 2.8% by weight ammonium hydroxide, with the balance being water.

#### Malodor Test Protocol

##### EXAMPLE 5

The half diaper of Example 3 containing either the neat malodor counteracting fragrance or the encapsulated fragrance were placed into an aluminum tray, approximately 5 cm deep, covered with a perforated aluminum sheet, in order to keep it out of view, up to the moment of the sniff-test. The sniff-test was performed in a "pre-ventilated" room by six graders. Each grader had been pre-selected for their sensitivity to the unpleasant smells present in an absorbent article after use and their ability to grade the unpleasantness of the odor in a consistent manner. Every grader evaluated the odor of each series of three products representing each sample using a pleasantness scale which ranges from 10 (most pleasant) to 1 (neutral rating). The pleasantness values for each sample were obtained as a mean of 18 observations (six graders, three products for each sample).

The "dry" products (simulating the conditions prior to soiling the product) containing either the powder or the neat oil were evaluated by a sniff-test method. The products were then wetted with 50 ml urine (reconstituted and synthetic), allowed to equilibrate for 10 minutes and evaluated. The wet products were then covered with a perforated aluminum sheet and were evaluated again after 150 minutes by a sniff-test method.

Test Results: Moisture Triggered ReleaseEXAMPLE 6

5        The spray dried multi component particles of examples 1 and 2, a reference sample comprising the neat fragrance, and the market product, were tested in order to show the malodor coverage capability of the controlled release system of the present invention.

10       The pleasantness grade values show significant differences between the product with the system and the reference samples.

TABLE 1

15	Sample	Dry Product		Wet Product	
		Reconstituted Urine		Synthetic Urine	
20	Example 1	1	5	6	
	Example 2	2	6	7	
25	Pamper (market product)	1	strong urine malodor		
	Reference sample (neat oil)	8	9	7	

30       These results show that the reference samples were not effective in counteracting the urine malodor. The samples comprising the multi component controlled release system of example

1 and 2 were successful in significantly reducing the noxious amine and ammonia odors and produce a pleasant odor. It can also be seen from the results that the products comprising the controlled release system of example 1 and 2 have very low odor intensity in the dry state. Thus, the controlled release system of the present invention sustains the volatile constituents of the malodor counteracting fragrance during storage, prior to the soiling of the product. High odor intensity is observed for the wetted products as a result of releasing the nano particle comprising the fragrance. Thus, the system of the present invention protect the volatile constituents of the fragrance and provides moisture triggered releases of a malodor counteracting fragrance upon need, in response to body fluids.

#### Test Results: Extended Malodor Coverage

##### EXAMPLE 9

The ability of the controlled release systems prepared in examples 1 and 2 and the reference samples to provide malodor coverage over an extended period of time, in the presence of body fluids, was tested.

The pleasantness grade values show significant differences between the product with the system and the reference samples.

TABLE 2

---

5	Sample	Wet Product 150 minutes	
		Reconstituted Urine	Synthetic Urine

---

	Example 1	7	7
10	Example 2	8	7

	Sample	strong urine malodor	
15	(market product)		

Reference Sample	2	1
(neat oil)		

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It can be seen from the results that the products comprising the controlled release system of example 1 and 2 have the ability to provide malodor coverage, in the presence of body fluids, over an extended period of time. The products comprising the controlled release system also show significant improvement over the performance of the current market product and over

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using the neat malodor counteracting fragrance.

What is claimed is:

1. A multi component moisture activated composition comprising:  
a plurality of solid nano particles, each of said solid nano particles comprising an  
5 effective amount of a first active agent.
2. The composition of claim 1 wherein said solid nano particles are formed of a  
hydrophobic material.
- 10 3. The composition of claim 2 wherein said hydrophobic material is selected from the  
group consisting of natural waxes and synthetic waxes, natural wax and silicon copolymers,  
synthetic wax and silicon copolymer, fatty acid esters, fatty alcohols, solid hydrogenated  
plant oils, natural polymers and synthetic polymers.
- 15 4. The composition of claim 2 wherein said hydrophobic material comprises carnauba  
wax.
5. The composition of claim 2 wherein said hydrophobic material comprises candelilla  
wax and silicon copolymer.
- 20 6. The composition of claim 1 further comprising a second active agent encapsulated in  
said moisture sensitive matrix wherein said matrix releases said second active agent upon  
contact with said moisture and continuously thereafter for an extended period of time.
- 25 7. The composition of claim 6 wherein said moisture sensitive matrix material is  
selected from the group consisting of a starch derivative, natural gum, polyvinyl alcohol,  
polysaccharide, protein, hydrocolloid, and mixtures thereof.
8. The composition of claim 7 wherein said hydrocolloid is selected from the group  
30 consisting of xanthan, maltodextrin, galactomanan, and tragacanth.

9. The composition of claim 7 wherein said natural gum is gum arabic.
10. The composition of claim 7 wherein said polyvinyl alcohol is present in an amount by weight of about 1% to about 70% by weight of said particle.
- 5 11. The composition of claim 10 wherein said polyvinyl alcohol is present in an amount by weight of about 70% to about 80% of said particle.
12. The composition of claim 11 wherein said extended period of time is up to about 48  
10 hours.
13. The composition of claim 10 wherein said polyvinyl alcohol has a degree of hydrolysis from about 75% to about 99%.
- 15 14. The composition of claim 7 wherein said polysaccharide is present in an amount by weight of about 1% to about 70% by weight of said particle.
15. The composition of claim 7 wherein said polysaccharide is present in an amount by weight of about 30% to about 40% of said particle and said polyvinyl alcohol is present in an  
20 amount by weight of about 30% to about 40% of said particle.
16. The composition of claim 15 wherein said composition releases an effective amount of said second active agent to provide a burst of said active agent.
- 25 17. The composition of claim 16 wherein after said burst of said agent said active agent is continuously released for an extended period of time.
18. The composition of claim 1 wherein said first active agent is a cosmetic composition or a pharmaceutical composition.

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19. The composition of claim 18 wherein said first active agent is selected from the group consisting of a moisturizing agent, fragrance, astringent, anti-inflammatory, anti-microbial and anti-fungal.
- 5 20. The composition of claim 1 wherein said first active agent is a malodor counteracting agent.
21. The composition of claim 1 wherein said first active agent comprises an antibacterial compound selected from the group consisting of cetyl pyridinium chloride, zinc chloride,  
10 chlorhexidine, quaternary ammonium compound, parabens, chitin, and a pH buffered material.
22. The composition of claim 20 wherein said first active agent is a biologically active agent having antiphlogistic property or an antibacterial property.
- 15 23. The composition of claim 22 wherein said malodor counteracting agent is bisabolol.
24. The composition of claim 20 wherein said malodor counteracting agent is a fragrance.
- 20 25. The composition of claim 6 wherein said second active agent is a cosmetic composition or a pharmaceutical composition.
26. The composition of claim 25 wherein said second active agent is selected from the group consisting of a moisturizing agent, fragrance, astringent, anti-inflammatory, anti-  
25 microbial and anti-fungal.
27. The composition of claim 6 wherein said second active agent is a malodor counteracting agent.
- 30 28. The composition of claim 6 wherein said second active agent comprises an antibacterial compound selected from the group consisting of cetyl pyridinium chloride, zinc

chloride, chlorhexidine, quaternary ammonium compound, parabens, chitin, and a pH buffered material.

29. The composition of claim 27 wherein said second active agent is a biologically active agent having antiphlogistic property or an antibacterial property.

30. The composition of claim 29 wherein said malodor counteracting agent is bisabolol

31. The composition of claim 27 wherein said malodor counteracting agent is a fragrance.

10

32. A method for producing the composition of claim 1 comprising the steps of:

- (i) incorporating said first active agent into said nano particles;
- (ii) forming an aqueous mixture comprising said nano particles and said water sensitive material; and

15 (iii) spray drying said mixture to form a dry powder composition.

33. The method of claim 32 wherein said aqueous mixture formed in step ii further comprises a second active agent.

20 34. A method for producing the composition of claim 6 comprising the steps of:

- (i) heating a hydrophobic material for forming said nano particles to a temperature above a melting point of said hydrophobic material to form a melt;
- (ii) dispersing said first active agent into said melt;
- (iii) dispersing said second active agent and said water sensitive matrix material in the aqueous phase;
- (iv) heating the dispersion to above the melting temperature of said hydrophobic material to form a hot melt;
- (v) mixing said hot melt with the aqueous phase to form a dispersion;
- (vi) homogenizing the dispersion at a temperature above the melting temperature until a homogeneous fine dispersion is obtained having a particle size of from about 1 micron to about 2 microns;

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(vii) cooling the homogenized dispersion to ambient temperature to form a suspension; and

(viii) spray drying the emulsified mixed suspension to form a dry powder composition.

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35. An absorbent article comprising a said moisture activated composition of claim 1

36. The absorbent article of claim 35 further comprising:  
a sanitary paper material.

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37. The absorbent article of claim 36 wherein said sanitary paper product is selected for the group consisting of a catamenial, sanitary napkin, pantiliner, diaper, and an adult incontinence garment.

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38. A method for providing malodor control over an extended period of time in a sanitary paper product comprising the step of providing a moisture activated malodor controlling composition, the malodor controlling composition comprising an effective amount of a malodor counteracting agent incorporated into a hydrophobic nano particle and encapsulated in a moisture sensitive matrix material.

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39. A method for delivering a pharmaceutical or cosmetic agent over an extended period of time in a sanitary paper product comprising the step of providing a moisture activated composition, said moisture activated composition comprising an effective amount of a cosmetic or pharmaceutical active agent incorporated into a hydrophobic nano particle encapsulated in a matrix material formed of a composition of polyvinyl alcohol or a composition of polyvinyl alcohol and polysaccharide.

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40. A controlled release composition comprising:  
a plurality of solid hydrophobic nano particles, each of said solid hydrophobic nano particles comprising an effective amount of an active agent.

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41. The composition of claim 40 wherein said hydrophobic material is selected from the group consisting of natural waxes and synthetic waxes, natural wax and silicon copolymers, synthetic wax and silicon copolymer, fatty acid esters, fatty alcohols, solid hydrogenated plant oils, natural polymers and synthetic polymers.

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42. The composition of claim 40 wherein said active agent is a cosmetic composition, pharmaceutical composition or malodor counteracting agent.

# INTERNATIONAL SEARCH REPORT

International application No.

PCT/US01/30084

## A. CLASSIFICATION OF SUBJECT MATTER

IPC(7) : A61L 9/00; A61F 13/15, 13/20; A61B 16/00

US CL : 424/76.11; 604/358, 359, 367

According to International Patent Classification (IPC) or to both national classification and IPC

## B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

U.S. : 424/76.11; 604/358, 359, 367

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)  
BRS

## C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	US 5,429,628 A (TRINH et al) 04 July 1995 (04.07.1995), see entire document.	1-22, 24-29, 31-42
Y	EP 0 908 174 A2 (INTERNATIONAL FLAVORS & FRAGRANCES INC.) 14 April 1999 (14.04.1999), see entire document.	1-22, 24-29, 31-42
Y	US 5,783,211 A (MANZO et al) 21 July 1998 (21.07.1998), see entire document.	23 and 30



Further documents are listed in the continuation of Box C.



See patent family annex.

\* Special categories of cited documents:

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